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Taguchi Approach For Diamond-like Carbon Film Processing

VijayKumar S. Jatti[a,*], Meena Laad[b], T.P.Singh[c]

[a]*Assistant Professor, Symbiosis Institute of Technology (SIT), Symbiosis International University (SIU), Lavale, Pune-412 115, Maharashtra State, India*
[b]*Associate Professor, Symbiosis Institute of Technology (SIT), Symbiosis International University (SIU), Lavale, Pune-412 115, Maharashtra State, India*
[c]*Director and Professor, Symbiosis Institute of Technology (SIT), Symbiosis International University (SIU), Lavale, Pune-412 115, Maharashtra State, India*

Abstract

In this study, Taguchi methodology is applied for depositing diamond-like carbon (DLC) films on P-type silicon substrates using inductively coupled plasma enhanced chemical vapor deposition (IC-PECVD) process. The Taguchi method is used to formulate the experimental layout, to analyze the effect of each process parameter and to predict the optimal choice for each process parameters such as bias voltage, bias frequency, deposition pressure and gas composition. It is found that these parameters have a significant influence on DLC properties such as I_D/I_G ratio, hardness and Young's modulus. The analysis of Taguchi method reveals that, in general the bias voltage significantly affects the I_D/I_G ratio, gas composition significantly affects the hardness and Young's modulus of DLC films. Experimental results are provided to verify this approach.

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Keywords: Diamond-like carbon;Plasma enhaced chemical vapor deposition;Taguchi approach;Analysis of variance

1. Introduction

Diamond-like carbon (DLC) films are primarily made of carbon atoms that are derived from carbon containing sources such as solid carbon targets, liquid and gaseous forms of hydrocarbons, Hainsworth and Uhure (2007). DLC films can be deposited by various types of deposition techniques and many people have studied the influence of different deposition parameters in a systematic manner, Erdemir and Donnet (2006). Generally, the effect of process parameters has been studied by changing one separate parameter at a time while keeping other parameters constant.

* Corresponding author. Tel.: +91-20-39116437; fax: +91-20-39116460.
E-mail address: vijaykumar.jatti@sitpune.edu.in

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This approach does not necessarily lead to a real optimum and provides no information about what happens when the factors are varied simultaneously. The method also requires an unnecessarily high number of runs. Experimental design provides an organized approach for coating optimization. It offers a means to study systems influenced by more than one parameter and gives better estimation of the variability and noise of the system and thus increases the reliability of the results. Experimental design is also a good way to minimize the number of depositions and tests required. Czyzniewski (2012), presented the results of investigations on the selection of optimal deposition parameters for WDLC coatings produced by pulsed reactive magnetron sputtering in order to obtain the coatings with high adhesion to the high speed steel and cemented carbide substrates. Pancielejko et al. (2012), determined the optimal values of selected deposition parameters of diamond-like-carbon coatings with the modified cathodic vacuum arc (MCVA) method. Therefore, a comprehensive study of the effects of process parameters (bias voltage, bias frequency, deposition pressure and gas composition) on the DLC film properties such as I_D/I_G ratio, hardness and young's modulus is of great significance. Although study of these parameters has been performed by many researchers, most of the studies do not consider both engineering philosophy namely design of experiments and mathematical formulation namely analysis of variance. Therefore, the Taguchi method, which is a powerful tool for parametric design of performance characteristics, is used to determine the optimal process parameters for minimum ID/IG ratio, maximum hardness and maximum Young's modulus in DLC processing.

2. Experimental Details

DLC films were deposited by using a standard inductively coupled plasma-enhanced chemical vapor deposition (IC-PECVD) reaction chamber powered by a 13.56 MHz RF power (50 W) source. A P-type silicon wafer (100) of 1.5 cm x 1.5 cm square area of 500 µm thickness was taken as substrate for depositing DLC film. The carbon source gas was methane (CH_4), diluent was hydrogen. The pressure inside the chamber was varied by closing and opening the throttle valve. The total flow rate was maintained a constant at 10 sccm, by varying the composition of the precursor gases. In this study, bias voltage, bias frequency, deposition pressure and gas composition were considered as deposition parameters. An L_9 (3^4) Taguchi orthogonal array was used as the experimental design. The parameters investigated and the levels were indicated in table 1.

Table 1. Deposition parameters and their levels

Level	Bias voltage (V)	Bias frequency (kHz)	Deposition pressure (µbar)	Gas composition (%)
1	-50	0.25	2	60:40
2	-100	6	4	80:20
3	-150	40	6	90:10

The Raman spectra of the coatings were obtained using Micro Raman spectrometer Labram 010 Model of DILOR-JOBIN-SPEX make. The source used was a He-Ne laser of wavelength 632 nm and 3 mW power. The obtained Raman spectra were fitted with two Gaussian curves after subtracting a linear background. The ratio I_D/I_G was obtained by computing the areas of D-peak and G-peak and taking their ratio.

The nano hardness was measured by Nano-hardness Tester (CSEM Instruments) fitted with an integrated optical (Nikon)/Atomic Force Microscope (Surface Imaging Systems) equipped with a Berkovich diamond indenter, having a triangular-pyramid shape. The reported hardness and Young's modulus are average values of 4 indentations for each sample. The hardness and modulus were determined by Oliver-Pharr method, Oliver and Pharr (1992).

3. Results and Discussions

Experiments are carried out with varying values of bias voltage, bias frequency, deposition pressure and gas composition. Table 2 shows the experimental parameters and observed values for I_D/I_G ratio, hardness and Young's modulus.

Table 2. L_9 (3^4) Taguchi orthogonal array and observed values

Factors				I_D/I_G ratio	S/N ratio	Hardness (GPa)	S/N ratio	Young's Modulus (GPa)	S/N ratio
Bias Voltage (V)	Bias Frequency (kHz)	Deposition Pressure (μbar)	Gas Composition (%)						
-50	0.25	2	60:40	0.11	19.1721	17.73	24.9742	186.74	45.4247
-50	6	4	80:20	0.1	20	16.79	24.501	189.94	45.5723
-50	40	6	90:10	0.3	10.4576	15.63	23.8792	173.86	44.804
-100	0.25	4	90:10	0.44	7.1309	16.57	24.3865	175.66	44.8935
-100	6	6	60:40	0.32	9.897	17.96	25.0861	191.22	45.6307
-100	40	2	80:20	0.33	9.6297	16.65	24.4283	172.81	44.7514
-150	0.25	6	80:20	0.74	2.6154	16.09	24.1311	185.46	45.365
-150	6	2	90:10	0.77	2.2702	16.88	24.5474	175	44.8608
-150	40	4	60:40	0.17	15.391	17.91	25.0619	188.59	45.5104

3.1. Raman spectroscopy

The Raman scattering is the inelastic scattering of photons by fundamental excitations in molecules and solids. It is used as a non-destructive technique for characterization of carbon based materials. DLC films are composed of amorphous carbon consisting of sp^2 and sp^3 contents. Raman spectroscopy characterizes the structural arrangement of carbon atoms in the film. The optical, electrical, mechanical and tribological properties of DLC films depend upon the ratio of the amount of sp^2 to that of sp^3 bonds. In the present study, Raman spectra of DLC films are observed. The obtained Raman spectra were fitted with two Gaussian curves after subtracting a linear background. The characteristic G peaks (graphite like sp^2 bonds) and D peaks (diamond like sp^3 bonds) were obtained for the DLC films. D peak is located at a wave number of 1311 cm^{-1} corresponding to sp^3 carbon bond and the G peak is located at a wave number of 1498 cm^{-1}. The ratio I_D/I_G was obtained by computing the areas below D-peak and G-peak and taking their ratio. It is observed that the with increasing sp^3/sp^2 ratio the graphite like sp^2-clusters become smaller in number and size and the I_D/I_G ratio is decreased, Irmer and Dorner-Reisel (2005). The Raman spectrum obtained by Raman spectroscopy for DLC film is shown in fig.1.

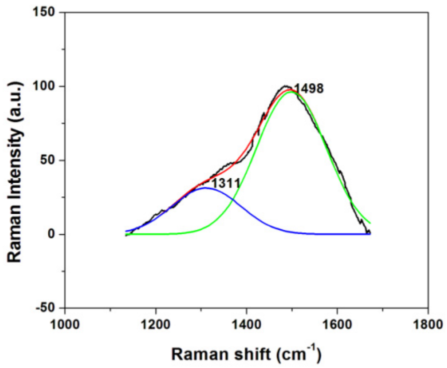

Fig.1. Raman spectrum of DLC film

3.2. Nano-hardness test

Hardness and Young's modulus were determined from the indentation load-displacement data. The load vs displacement curve obtained for DLC film is illustrated in fig.2. The reported hardness and Young's modulus are average values of 4 indentations (a, b, c and d) on each sample. The loading and unloading rate was same with a value of 10 mN/min. A maximum load of 5 mN and the maximum depth of indentation of 125 nm were applied over 60 seconds. Hence, total deformation was confined within the film confirming the fact that the hardness and Young's modulus are obtained from the films only, i.e. there was no substrate effect, Erdemir and Donnet (2006). The residual displacement was 50 nm for a total displacement of 125 nm. Thus, the film underwent 60% elastic deformation and 40% plastic deformation.

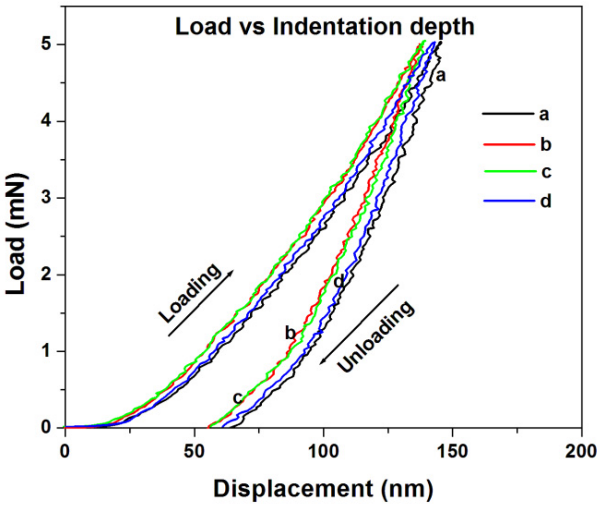

Fig.2. Load-displacement curve of DLC film

3.3. Analysis of Signal to noise ratio

In the Taguchi method, the term 'signal' represents the desirable value (mean) for the output characteristic and the term 'noise' represents the undesirable value for the output characteristic. Taguchi uses the S/N ratio to measure the quality characteristic deviating from the desired value. There are several S/N ratios available depending on type of characteristic: smaller is better, nominal is best and larger is better, Roy (2001). In this study, smaller is better S/N ratio was used for I_D/I_G ratio, larger is better S/N ratio was used for hardness and young's modulus. The S/N ratio values calculated for I_D/I_G ratio, hardness and Young's modulus were listed in table 2. Fig.3 to fig.5. shows the main effects plot for S/N ratios. The level of a factor with the highest S/N ratio was the optimum level for responses measured. From the S/N ratio analysis in figures 3 to 5, the optimal deposition conditions were -50 V bias voltage, 40 kHz bias frequency, 4 μbar deposition pressure and 60:40 gas composition to achieve minimum I_D/I_G ratio, -100 V bias voltage, 6 kHz bias frequency, 4 μbar deposition pressure and 60:40 gas composition to achieve maximum hardness, -50 V bias voltage, 6 kHz bias frequency, 4 μbar deposition pressure and 60:40 gas composition to achieve maximum Young's modulus.

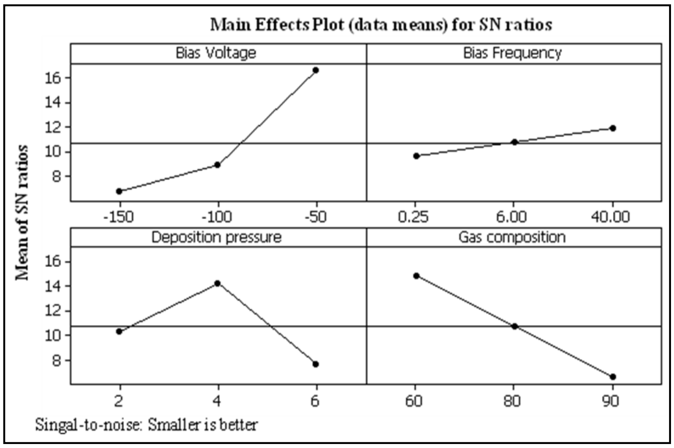

Fig.3. Mean effect plot for SN ratios of I_D/I_G ratio

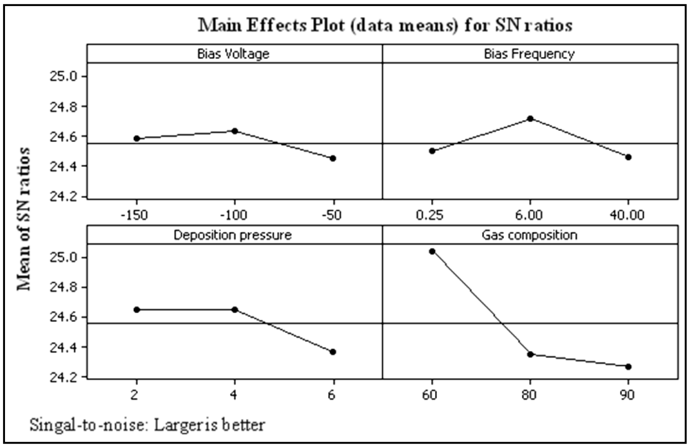

Fig.4. Mean effect plot for SN ratios of hardness

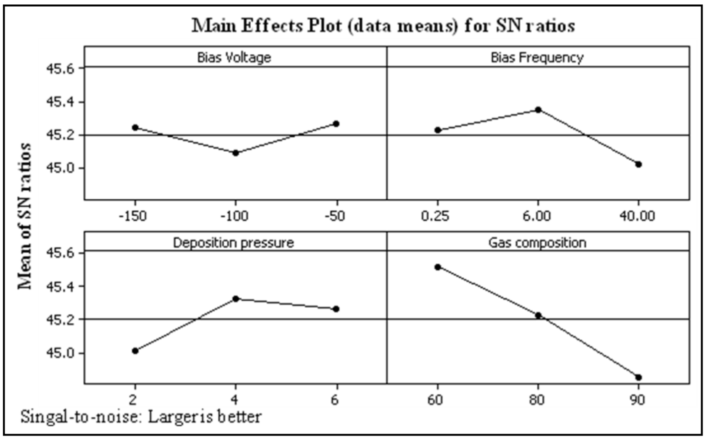

Fig.5. Mean effect plot for SN ratios of Young’s modulus

3.4. Analysis of variance

ANOVA was used to determine the significant parameters influencing the I_D/I_G ratio, hardness and Young's modulus of DLC coating. From ANOVA of I_D/I_G ratio, it was found that bias voltage is most significant factor with a contribution of 46.46 % followed by gas composition with a contribution of 28.7 % that influence the I_D/I_G ratio. From ANOVA of hardness, it was found that gas composition is most significant factor with a contribution of 78.2 % that influence the hardness of DLC film. From ANOVA of Young's modulus, it was found that gas composition is most significant factor with a contribution of 63.57 % that influence the Young's modulus of DLC film.

Table 3. Analysis of variance for I_D/I_G ratio

Factor	Degree of Freedom	Average S/N Values			Sum of squares	Mean square	Percentage of Contribution (%)
		Level 1	Level 2	Level 3			
Bias Voltage	2	6.759	8.886	16.543	0.2282	0.114	46.46
Bias Frequency	2	9.639	10.722	11.826	0.0447	0.0223	9.099
Deposition Pressure	2	10.357	14.174	7.657	0.0772	0.0386	15.72
Gas Composition	2	14.82	10.748	6.62	0.1409	0.0705	28.7
Error	0						
Total	8				0.491		100

Table 4. Analysis of variance for hardness

Factor	Degree of Freedom	Average S/N Values			Sum of squares	Mean square	Percentage of Contribution (%)
		Level 1	Level 2	Level 3			
Bias Voltage	2	24.58	24.63	24.45	0.1871	0.0935	3.543
Bias Frequency	2	24.5	24.71	24.46	0.4057	0.2028	7.676
Deposition Pressure	2	24.65	24.65	24.37	0.5583	0.2791	10.56
Gas Composition	2	25.04	24.35	24.27	4.1331	2.0665	78.217
Error	0						
Total	8				5.2842		100

Table 5. Analysis of variance for Young's Modulus

Factor	Degree of Freedom	Average S/N Values			Sum of squares	Mean square	Percentage of Contribution (%)
		Level 1	Level 2	Level 3			
Bias Voltage	2	45.25	45.09	45.27	23.061	11.531	4.95
Bias Frequency	2	45.23	45.35	45.02	73.829	36.914	15.855
Deposition Pressure	2	45.01	45.33	45.27	72.748	36.374	15.623
Gas Composition	2	45.52	45.23	44.85	296.01	148.005	63.57
Error	0						
Total	8				465.648		100

3.5. Confirmation experiments

In order to validate the results obtained, three confirmation experiments were conducted for each of the response characteristics (I_D/I_G ratio, hardness and Young's modulus) at optimal level of process variables. The average values of the characteristics were obtained and compared with the predicted values as tabulated in table 6.

Table 6. Validation experiment results based on Taguchi optimal parameter setting

Optimal values	I_D/I_G ratio	Hardness (GPa)	Young's modulus (GPa)
-50 V, 40 Khz , 4 μbar, 60:40	0.18	–	–
-100 V, 6 Khz, 4 μbar, 60:40	–	18.29	–
-50 V, 6 Khz, 4 μbar, 60:40	–	–	192.61

4. Conclusions

This study discussed an application of Taguchi experimental method for investigating the influence of deposition parameters on the I_D/I_G ratio, hardness and Young's modulus during deposition of DLC films. The level of importance of the deposition parameters on the I_D/I_G ratio, hardness and Young's modulus was determined by ANOVA. The optimal condition for I_D/I_G ratio was -50 V bias voltage, 40 kHz bias frequency, 4 μbar deposition pressure and 60:40 gas composition. The optimal condition for Hardness was -100 V bias voltage, 6 kHz bias frequency, 4 μbar deposition pressure and 60:40 gas composition. The optimal condition for Young's Modulus was -50 V bias voltage, 6 kHz bias frequency, 4 μbar deposition pressure and 60:40 gas composition. It was found that bias voltage has greater influence on I_D/I_G ratio and gas composition has greater influence on hardness and young's modulus. Results showed that Taguchi method is a powerful tool for providing experimental diagrams and to perform the experiments efficiently and economically. This establishes the reliability of Taguchi method as one of the most accurate optimization approach.

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